

BROWN & WILLIAMSON TOBACCO CORPORATION

Research Department

Standard Method of Analysis

Method No.: SM-134

Date Issued: March 11, 1983

THE AUTOMATED DETERMINATION OF
TAR, NICOTINE, AND CARBON MONOXIDE BY THE FTC METHOD

Mr. R. A. Burge

Distribution

Dr. J. H. Lauterbach

Mr. J. F. Nall

Mr. L. L. Shockley

Mr. M. S. Frank

Mr. R. A. Burge

Library (2)

Greens

2023100237

BROWN & WILLIAMSON TOBACCO CORPORATION

Research Department

Standard Method of Analysis

Method No.: SM-134 Date Issued: March 11, 1983

Method Class:

Title: The Automated Determination of Tar, Nicotine, and Carbon Monoxide by the FTC Method

Author: Mr. R. A. Burge

Summary: Cigarettes are smoked on a Phipps & Bird smoking machine. Carbon monoxide (CO) is collected and measured. Gelman pads are used to trap particulates and the total particulate matter (WTPM) is measured by weight. The Gelman pads and filter tips are placed into vials and extracted with isopropanol. Water and nicotine content of the pad extracts and the nicotine content of the filter extracts are determined. Tar is then calculated from these results. The method measures WTPM, water, nicotine, CO and puff number. Tar is calculated using WTPM, water and nicotine results. Nicotine Retention Efficiencies (NRE) are calculated using nicotine results. Tar, nicotine, and CO are reported as mg/cig.

Sample Size: The sample consists of 80 cigarettes divided into 16 subsamples (ports) of five cigarettes each. Each of the subsamples is smoked onto a filter trap where the WTPM is collected and measured.

Range and/or Sensitivity: Tar can be measured precisely in samples delivering from 0.5 - 40.0 mg/cig. CO can be measured in samples delivering 0.5 - 25.0 mg/cig.

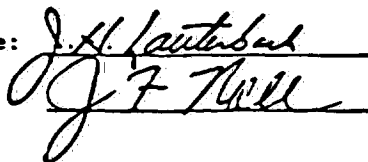
Precision: Method precision was estimated for tar from smoke determinations on two control samples (8.8 and 5.0 mg/cig). The standard deviations were 0.5 and 0.4 mg/cig, respectively. The relative standard deviations were 5.7 and 8.0%, respectively.

Analysis Time - Setup: (man-hours) - 8 Hours (16 port sample)

- Take-down: (man-hours) - 2.0

- Variable: Approximately 20 ± 2 samples/day.
(man-hours/sample) = 4.0

Approved for issue:

Date: 3/11/83Date: 3/16/83

2023100238

1. Principle of Method

Total particulate matter (WTPM) and puff number are measured by smoking cigarettes on a Phipps & Bird Mark I smoke machine (ref. 1). The WTPM is collected on a smoke trap that is used to collect aerosols. The trap (Gelman pad), which collects smoke particulates, is extracted with isopropanol containing two (2) internal standards. Nicotine and water are measured in this extract. Nicotine is determined using an AutoAnalyzer I system (ref. 2). Water is determined using gas chromatography (ref. 3). Filter tips are extracted with isopropanol, and the extract is analyzed for nicotine via an AutoAnalyzer I system (ref. 2).

Carbon monoxide is determined in the gas phase of cigarette smoke by nondispersive infrared (NDIR) analyzer. The gas phase of each port of smoke is collected in a bag and each bag is subsequently sampled when smoking is complete.

Tar and CO are calculated using a computer system. Results are reported as mg/cig.

2. Range and Sample Size

- 2.1 This method is used to determine tar, nicotine, WTPM, CO, % NRE, and puff number in whole smoke. A minimum of 20 cigarettes is recommended. Five cigarettes (one port) are smoked per Gelman pad (smoke trap). Two pads are combined and extracted with isopropanol. These extracts are used to measure nicotine and water which are used to calculate tar. % NRE is calculated from the nicotine extract results. CO is measured in the vapor phase. (Recommended minimum ports smoked = 4; normal = 16.)
- 2.2 Tar content is determined routinely from 0.5 - 40.0 mg/cig.
- 2.3 Nicotine content is measured routinely from 0.02 - 2.6 mg/cig using AutoAnalyzer Methodology. Higher concentrations are measured with appropriate dilutions. Nicotine levels as low as 0.001 mg/cig are measured via gas chromatography method (ref. 3).

2023100239

- 2.4 CO is measured routinely from 0.5 - 20.0 mg/cig. Higher concentrations can be measured, but modifications in the smoking apparatus are needed.
 - 2.5 Water content is measured routinely from 0.2 - 4.0 mg/cig. (0.1 - 2.0 mg/mL). Higher concentrations are measured with appropriate dilutions.
 - 2.6 Puff number is automatically calculated by the computer using a pulse generator as a signal. Typical ranges for this result are from 6.0 - 15.0 puffs/cig. % NRE obtained from nicotine is also calculated by the computer. The typical range for % NRE is 25 - 80%.
3. Safety Precautions
- 3.1 Normal safety precautions are employed.
 - 3.2 Nicotine is very poisonous. Standards containing pure nicotine should be handled with caution (ref. 2).
 - 3.3 CO is a very poisonous gas, therefore CO cylinders must be stored in a hood. Calibration standards should be handled with caution.
4. Interferences and Corrective Measurements
- 4.1 Plain end cigarettes are taped so that leakage does not occur when these cigarettes are inserted into the dental dam (rubber membrane) on the front of the Gelman pad holder.
 - 4.2 Charcoal filters cannot be analyzed for nicotine by the AutoAnalyzer method. Charcoal interferes with this method. GC analysis is used to determine nicotine concentration for these filters.
 - 4.3 Appropriate measures to insure the correct computer response must be taken if less than five cigarettes are smoked per Gelman pad (see computer manual for necessary corrective steps).
- CAUTION: CO measurements are not valid for less than five cigarettes/port, unless appropriate volume corrections are made.

2023100240

- 4.4 Special smoke delivery methods must be instituted if tar results are expected to be below 0.5 mg/cig (refs. 4, 5).
- 4.5 Care must be taken when smoking cigarettes that have ventilated tipping. The vent holes in the tipping must not be covered by the rubber membrane on the end of the smoke trap assembly.
- 4.6 For cigarettes with filters that have been pulled, the tipping on tobacco sections containing ventilation must be taped or blocked so that ventilation does not occur.
- 4.7 When tobacco sections of cigarettes are smoked, they must be taped with "Magic" tape so that leakage does not occur when these cigarettes are inserted into the dental dam (rubber membrane) on the front of the Gelman pad holder.

5. Precision

- 5.1 Tar deliveries of two control samples were collected at the rate of 6 ports/day for a ten day period.
- 5.2 The mean (\bar{X}), standard deviation (S.D.), and percent relative standard deviation (R.S.D.) of the tar deliveries for both referees were:

	<u>Control 1</u>	<u>Control 2</u>
\bar{X}	8.8 mg/cig	5.0 mg/cig
S.D.	0.5	0.4
R.S.D.	5.7%	8.0%
N	30	30

- 5.3 Nicotine deliveries of two referee samples (BCR-2 and VRL-1) were collected for 13 days. Each referee sample was run from 2 to 10 times each day.
- 5.4 The mean (\bar{X}), standard deviation (S.D.), and percent relative standard deviation (R.S.D.) of the nicotine deliveries for both referees were:

	<u>BCR-2</u>	<u>VRL-1</u>
\bar{X}	0.28 mg/cig	0.78 mg/cig
S.D.	0.04	0.05
R.S.D.	14%	6%
N	81	95

2023100241

- 5.5 CO deliveries of two control samples were collected at the rate of 8 ports/day for a ten day period.
- 5.6 The mean (\bar{X}), standard deviation (S.D.), and percent relative standard deviation (R.S.D.) of the CO deliveries for the two control samples were:

	<u>Control 3</u>	<u>Control 4</u>
\bar{X}	8.8 mg/cig	3.3 mg/cig
S.D.	0.76	0.33
R.S.D.	7.2%	10.0%
N	80	80

6. Apparatus

- 6.1 Phipps & Bird Mark I Smoking Machine, designed and licensed by Philip Morris Research Laboratories, with a 20 port capacity.
- 6.2 Gelman smoke analysis filters - plastic disposable type holders. This filter holder is leak proof and chemically inert. It meets or exceeds all I.S.O./D.I.S 3308.2 standards.
- 6.3 Filter discs made of a glass fiber material, 1 - 2 mm thick with a diameter of 47 mm. The rough filter surface faces the oncoming smoke. The filter material must have the following characteristics.
- 6.3.1 It must retain at least 99.9% of all particulates having a diameter equal to or greater than $0.3 \mu\text{m}$ of a dioctylphthalate aerosol at a linear air velocity of 140 mm/s (28 linear feet/minute (LF/min)).
- 6.3.2 The draw resistance of a filter assembly must not exceed 900Pa (9 mbar) at a flow of 17.5 cc/s.
- 6.3.3 The polyacrylate binder shall not exceed 5% (m/m).
- 6.4 Thin latex rubber membrane made from dental dam cut into square pieces approximately 3.5 x 3.5 cm. This square has a hole in the center made from a #1 hole punch.

2023100242

- 6.5 Silicone rubber "O" rings, small - 3/8" I.D.
- 6.6 Alnor Velometer Type 3002: Used to measure the machine airflow at each of the three tri-ducts. A jet probe, #3910, to measure the air velocity is connected to the Alnor Velometer. Each duct is set to pull between 350 and 500 LF/min air velocity.
- 6.7 Standard Electric Timer Model 600-100: Used to measure the puff duration to 1/100th of a second.
- 6.8 A 50 cc Buret or Soap Bubble Flow Meter: Used to measure the 35 cc puff volume for each of the 20 ports. It is graduated in 0.1 cc intervals.
- 6.9 Analytical Balance - Mettler Model HL-32: 160 g capacity, accurate to 0.1 mg.
- 6.10 Filtrona Model ATCOM 300 CK Carbon Monoxide NDIR Analyzer: Equipped with adapter unit for the Phipps & Bird Model Mark I smoking machine.
- 6.11 Computer Hardware:
 - 6.11.1 Hewlett-Packard 3070A Terminal
 - 6.11.2 Hewlett-Packard 2240A Control Processor
 - 6.11.3 Hewlett-Packard 1000 E Computer
- 6.12 Vertical Vester Shaker
- 6.13 Filamatic vial filler set at 20 cc delivery.
- 6.14 Thread - 325 yd spool of J. & P. Coats black, cotton covered, polyester dual duty sewing thread.
- 6.15 Butt Marker
- 6.16 Set of Class S NBS calibrated weights.

7. Chemicals

- 7.1 Hi-Vacuum Pump Oil
- 7.2 SNOOP
- 7.3 CO Standard Gas Mixtures supplied by AIRCO or Matheson.
 - 7.3.1 6.0% CO, 94.0% high purity nitrogen.
 - 7.3.2 2.0% CO, 98.0% high purity nitrogen.
 - 7.3.3 4.0% CO, 96.0% high purity nitrogen.

CAUTION: These tanks should be stored in a well ventilated area such as a fume hood due to the poisonous nature of CO.

2023100243

8. Procedure

- 8.1 Sample Preparation - Place 30 to 60 randomized packs (600 - 1200 cigarettes) in a composite pool (normal sample size).**
- 8.1.1 Randomly select 80 cigarettes from the composite sample pool and butt mark.**
- 8.1.2 Normal butt markings are:**
- Filtered Cigarettes - tipping plus 3 mm.**
- Plain End Cigarettes - 23 mm.**
- 8.1.3 Place a filter insertion mark on the filter tipping 11 mm from the end of the filter. This mark designates how far the filter end of the cigarette is inserted into the dental dam on the smoke trap holder (8 mm insertion into dental dam plus 3 mm for plastic lucite bar on Phipps & Bird smoke machine). Plain end cigarettes carry the same insertion mark.**
- 8.1.4 Place the 80 cigarettes into a plastic Tupperware container, which is then capped, computer labeled, and delivered to the smoke laboratory.**
- 8.1.5 Condition the cigarettes in the Tupperware container (uncapped) for at least 16 hours at $75 \pm 2^\circ\text{F}$ and $60 \pm 2\%$ R.H. Typical moisture level in the tobacco after conditioning is approximately 13.5%.**
- 8.2 Smoking Machine Setup, Calibration, and Checkout**
- Check the smoke machine each day as follows:**
- 8.2.1 Check the syringes for oil content. They should contain at least 2 cc of oil (Hi-Vacuum Pump Oil) to lubricate and leak seal the plungers. To add oil, unscrew the syringe by hand, remove plunger and add oil to the large open end of the syringe while keeping a finger over the small threaded end. Replace the plunger and reconnect the syringe, being careful not to break the metal tip off of the syringe when tightening.**

2023100244

- 8.2.2 Turn the main power switch to ON. Turn the power switch on the console to ON (this powers the plug strip in the smoke machine). Set the normal/volume check switch to NORMAL. Set the clock switch to AUTOMATIC. Allow the machine to cycle at one minute intervals for 10 - 15 minutes. While the machine is cycling, measure the one minute time intervals with a stop watch. The time of each cycle should be one minute ± 0.05 seconds.
- 8.2.3 Volume check two different reference ports each morning (e.g. - a combination of ports such as 1&11, 2&12, 3&13, ... 10&20). After completion of the smoke schedule for the day, volume check all ports and reset to a 35 ± 0.3 cc volume if necessary. Volume checking of each port is accomplished as follows: using an inverted 50 cc buret and SNOOP, draw the solution up to the 40 cc mark to lubricate the inner surface of the buret. Turn the normal/volume check switch to VOLUME CHECK and the clock switch to MANUAL. Using a single bubble, adjust to approximately 0.5 cc on the buret and insert the tube from the buret into the port receiver. Press the manual trip switch on the cord extending from the Filamatic unit in the base of the smoking machine. Subtract the original bubble reading from the final reading obtained after pressing the switch on the cord. The volume measurement for each port should be 35 ± 0.3 cc. Make necessary adjustments to any port that is outside these limits. This is accomplished by loosening the set screw on the eccentric cam located next to the base of the syringe on the lower cam shaft. Move the cam up or down as required and lock the set screw. Measure the puff volume again as above.

2023100245

- 8.2.4 The standard puff duration is 2.0 ± 0.05 seconds for an individual puff. Use a standard automatic timing device to measure puff duration. This device plugs into the timing circuit located on the upper right hand corner of the smoke machine cabinet.
- 8.2.5 Check the puff counters to see that ten units (10.0) are registered with each puff; if necessary, adjust the pulse generator located in the rear of the puff counter cabinet so that each counter registers 10.0 count units.
- 8.2.6 Turn the normal/volume switch to NORMAL.
- 8.2.7 Check the air velocity in the tri-duct hood system using the ALNOR velometer and test probe. Each tri-duct should be set at 400 ± 50 LF/min.
- 8.2.8 Adjust the velocity to 400 LF/min at each duct. After adjusting each duct, by using the vane above the duct hole, read the velocity again to insure the proper air flow across all smoking machines.
- 8.3 Preparation of Smoke Assembly Traps.
 - 8.3.1 Use preassembled Gelman disposable filters.
 - 8.3.2 Leak test the assembly before use by subjecting it to a vacuum equivalent to 9.0 inches of water. Discard all assemblies that do not hold this vacuum.
 - 8.3.3 Fasten a piece of dental dam, 35 mm x 35 mm square with a 4-6 mm diameter hole in the center (cut with a #1 hole punch), to the front of the Gelman holder with an "O" ring. Care is taken to assure that the hole is centered and not stretched out of proportion.
 - 8.3.4 Place the assembly into its proper position in the smoke machine tray (ten per tray).

2023100246